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Supplementary Information

Highly Reactive meso-Like Positions of Dipyrihexaphyrin

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3,5-Bis(5-carboethoxy-3,4-diethyl-2-pyrryl)pyridine

3,5-Bis(3,4-diethyl-2-pyrryl)pyridine (**4b**)

Di-2,6-pyrihexaphyrin (**5a**)

Di-3,5-pyrihexaphyrin (**5b**)

meso-Methoxydihydrodi-2,6-pyrihexaphyrin (**6a**)

meso-Cyanodihydrodi-2,6-pyrihexaphyrin (**7a**)

meso-Bis(butylamino)tetrahydrodi-2,6-pyrihexaphyrin (**8a**)

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1. Synthesis and characterization data

General

Solvents were purified prior to use by conventional methods. $CDCl_3$ was passed through basic Al_2O_3 before use. Other chemicals were of reagent grade and used as received. Wakogel C-300 silica gel (Wako Junyaku) was used for column chromatography. ^{1}H NMR was recorded on a Varian Inova 400 spectrometer. Chemical shifts were referenced with respect to $(CH_3)_4Si$ (0 ppm) as an internal standard. ESI-MS spectra were measured with a Mariner PE Biosystems using methanol or acetone as solvent. The UV-visible spectra were measured on a JASCO V-570 spectrometer. Elemental analyses of C, H, and N were made with a YANACO MT-5 CHN recorder.

3,5-Bis(5-carboethoxy-3,4-diethyl-2-pyrryl)pyridine

To a mixture of 3,5-dibromopyridine (940 mg, 4.0 mmol), 5-carboethoxy-3,4-diethyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxoborolan-2-yl)pyrrole (ca. 8.4 mmol), $Pd(OAc)_2$ (22 mg, 0.10 mmol), and triphenylphophine (47 mg, 0.18 mmol), were added aqueous solution (9.0 ml) of potassium carbonate (1.7 g, 12 mmol), and DMF (30 ml). The reaction mixture was heated at 90~100˚C with stirring under argon for 24 h. After cooling, the reaction mixture was partitioned between CH_2Cl_2 and water. The organic products were extracted from the water layer with CH_2Cl_2. The combined organic layer was dried over anhydrous Na_2SO_4 and then evaporated to dryness under vacuum. The residue was crystallized from methanol to afford white powders (1.33 g) of the cross coupling product. Yield 72%.

^{1}H NMR (400 MHz, δ-value in $CDCl_3$) 9.08 (br, 2H, N*H*); 8.67 (d, 2H, J=2.3 Hz, pyridine-α-*H*); 7.83 (t, 1H, J=2.3 Hz, pyridine-γ-*H*); 4.34 (q, 4H, J=7.2 Hz, OC*H*$_2$Me); 2.80, 2.60 (q×2, 4H×2, J=8.6 Hz, C*H*$_2$Me); 1.36 (t, 6H, J=7.2 Hz, OCH$_2$*Me*); 1.22, 1.18 (t×2, 6H×2, J=7.6 Hz, CH$_2$*Me*). ESI-MS 466.23/466.27 (found/calcd for $C_{27}H_{35}N_3O_4$+H$^+$). Analysis calcd. for $C_{27}H_{35}N_3O_4$: C, 69.65; H, 7.58; N, 9.03. Found: C, 69.03; H, 7.61; N, 9.10.

3,5-Bis(3,4-diethyl-2-pyrryl)pyridine (**4b**)

Degassed ethyleneglycol (22.6 ml) was added to a mixture of 3,5-bis(5-carboethoxy-3,4-diethyl-2-pyrryl)pyridine (1.33 g, 2.86 mmol) and NaOH (1.14 g, 28.5 mmol) under argon, and the whole mixture was heated with stirring at 190 ˚C for 2 h. After cooling, water was added to the cooled solution to form yellow powders. The formed precipitates were filtered, washed with water, and then dried to give 869 mg of the product. Yield 94%.

^{1}H NMR (400 MHz, δ-value in $CDCl_3$) 8.51 (d, 2H, J=2.1 Hz, pyridine-α-*H*); 8.09 (br, 2H, N*H*); 7.71 (t, 1H, J=2.1 Hz, pyridine-γ-*H*); 6.69 (s, 2H, pyrrole-α-*H*); 2.63, 2.54 (q×2, 4H×2, J=8.2 Hz, C*H*$_2$Me); 1.27, 1.20 (t×2, 6H×2, J=7.5 Hz, CH$_2$*Me*). ESI-MS 322.25/322.23 (found/calcd for $C_{21}H_{27}N_3$+H$^+$).

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Analysis calcd. for $C_{21}H_{27}N_3$•(H_2O): C, 74.30; H, 8.61; N, 12.38. Found: C, 75.49; H, 8.26; N, 12.14.

Di-2,6-pyrihexaphyrin (**5a**)

To 2,6-bis(3,4-diethyl-2-pyrryl)pyridine (**4a**) (100 mg, 0.31 mmol) were added CH_2Cl_2 (63 ml), boron trifluoride diethyl etherate (0.52 mL, 4.12 mmol, 13.3 eq.), and 37% aqueous formaldehyde solution (26.7 μL, 0.35 mmol, 1.1 eq.) under argon. After the mixture was refluxed with stirring for 22 h, 2,3-dichloro-5,6-dicyanoquinone (DDQ) (85 mg, 0.37 mmol, 1.2 eq.) was then added. The reaction mixture was stirred for 30 min at ambient temprature. The resulting solution was washed with aqueous K_2CO_3 solution. The organic layer was dried over Na_2SO_4, and then evaporated to dryness and the residue was recrystallizated from CH_2Cl_2-hexane to give orange powders (81.5 mg). Yield 79%.

^{1}H NMR (400 MHz, δ-value in $CDCl_3$) 7.74 (t, 2H, J=7.9 Hz, pyridine-γ-*H*); 7.35 (d, 4H, J=7.9 Hz, pyridine-β-*H*); 6.93 (s, 2H, meso-*H)*; 2.67, 2.55 (q×2, 8H×2, J= 8.5 Hz, C*H*$_2$Me); 1.23, 1.07 (t×2, 12H×2, J=7.7 Hz, CH_2*Me*). UV-vis (λ_{max} (log ε) in CH_2Cl_2) 293 (4.43), 332 (4.21), 453 (4.75), 516 (sh, 3.94) nm. ESI-MS 663.40/663.42 (found/calcd for $C_{44}H_{50}N_6$+H^+). Analysis calcd. for $C_{44}H_{50}N_6$•(H_2O)$_2$: C, 75.61; H, 7.79; N, 12.02. Found C, 74.46; H, 7.49; N, 12.55.

Di-3,5-pyrihexaphyrin (**5b**)

To 3,5-bis(3,4-diethyl-2-pyrryl)pyridine (**4b**) (50 mg, 0.16 mmol) were added dry CH_2Cl_2 (32 ml), trifluoroacetic acid (0.24 mL, 1.9 mmol, 12.2 eq.), and 37% aqueous formaldehyde solution (14 μL, 0.18 mmol, 1.2 eq.) under argon. After the mixture was stirred for 22 h at ambient temperature, DDQ (42.5 mg, 0.19 mmol, 1.2 eq.) was then added. The reaction mixture was stirred for 30 min at ambient temprature. The resulting solution was neutralized with aqueous K_2CO_3 solution. The organic layer was separated, dried over Na_2SO_4, and then purified by column chromatography on alumina. The red fraction eluted with ethyl acetate was evaporated to dryness and the residue was recrystallizated from CH_2Cl_2-hexane to give red powder (16.6 mg). Yield 32%.

^{1}H NMR (400 MHz, δ-value in $CDCl_3$) 8.75 (d, 4H, J=2.0 Hz, pyridine-α-*H*); 8.25 (t, 2H, J=2.0 Hz, pyridine-γ-*H*); 6.92 (s, 2H, meso-*H)*; 2.71, 2.61 (q×2, 8H×2, J=8.5 Hz, C*H*$_2$Me); 1.26, 1.13 (t×2, 12H×2, J=7.7 Hz, CH_2*Me*). UV-vis (λ_{max} (log ε) in CH_2Cl_2) 294 (4.59), 458 (4.82), 520 (4.09) nm. ESI-MS 663.45/663.42 (found/calcd for $C_{44}H_{50}N_6$+H^+). Analysis calcd. for $C_{44}H_{50}N_6$•(H_2O)$_2$(CH_2Cl_2)$_2$: C, 63.59; H, 6.73; N, 9.67. Found: C, 63.69; H, 6.69; N, 9.70.

meso-Methoxydihydrodi-2,6-pyrihexaphyrin (**6a**)

Di-2,6-pyrihexaphyrin (**5a**) (16.1 mg, 24.3 μmol) was dissolved in a mixture of CH_2Cl_2 (5 ml) and methanol (3 mL, 75 mmol). After stirring for 2 h at room temperature, this solution was evaporated to dryness and the residue was recrystallizated from CH_2Cl_2-MeOH to give red powder (13.0 mg). Yield 77%.

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^{1}H NMR (400 MHz, δ-value in $CDCl_3$) 11.35 (br, 2H, N*H*); 7.62 (t, 2H, J=7.9 Hz, pyridine-γ-*H*); 7.38, 7.19 (d×2, 2H×2, J=7.9 Hz, pyridine-β-*H*); 7.04, 5.47 (s×2, 1H×2, meso-*H*); 3.34 (s, 3H, O*Me*); 2.72, 2.58 (m×2, 8H×2, CH_2Me); 1.27, 1.20, 1.16, 1.15 (t×4, 6H×4, J=7.5 Hz, CH_2*Me*). UV-vis (λ_{max} (log ε) in CH_2Cl_2) 296 (4.52), 330 (4.51), 462 (sh, 4.25), 505 (4.35) nm. ESI-MS 695.50/695.44 (found/calcd for $C_{45}H_{54}N_6O+H^+$). Analysis calcd. for $C_{45}H_{54}N_6O•(CH_2Cl_2)$: C, 70.84; H, 7.24; N, 10.78. Found: C, 70.64; H, 7.58; N, 10.59.

meso-Cyanodihydrodi-2,6-pyrihexaphyrin (**7a**)

To di-2,6-pyrihexaphyrin (**5a**) (15.5 mg, 23.4 μmol) dissolved in CH_2Cl_2 (5 ml) were added an aqueous solution (0.5 ml) of sodium cyanide (3.5 mg, 0.071 mmol, 3.1 eq.) and acetic acid (0.08 mL). The reaction mixture was stirred for 3 h at room temprature. The resulting solution was then washed with aqueous K_2CO_3 solution. The organic layer was separated, dried over Na_2SO_4, and then evaporated to dryness. The residue was recrystallizated from CH_2Cl_2-hexane to give red powder (12.7 mg). Yield 79 %.

^{1}H NMR (400 MHz, δ-value in $CDCl_3$) 11.85 (br, 2H, N*H*); 7.65 (t, 2H, J=7.9 Hz, pyridine-γ-*H*); 7.40, 7.25 (d×2, 2H×2, J=7.9 Hz, pyridine-β-*H*); 7.05, 5.32 (s×2, 1H×2, meso-*H*); 2.8~2.6 (m, 12H, CH_2Me); 2.51 (q, 4H, 8.4 Hz, CH_2Me); 1.27, 1.19, 1.14, 1.13 (t×4, 6H×4, J=7.6 Hz, CH_2*Me*). UV-vis (λ_{max} (log ε) in CH_2Cl_2) 296 (4.66), 334 (4.61), 461 (sh, 4.28), 516 (4.52) nm. ESI-MS 690.40/690.43 (found/calcd for $C_{45}H_{51}N_7+H^+$). Analysis calcd. for $C_{45}H_{51}N_7•(H_2O)_2$: C, 74.45; H, 7.64; N, 13.51. Found: C, 74.22; H, 7.45; N, 13.25.

meso-Bis(butylamino)tetrahydrodi-2,6-pyrihexaphyrin (**8a**)

To di-2,6-pyrihexaphyrin (**5a**) (11.2 mg, 16.9 μmol) in dry CH_2Cl_2 (5 ml) was added butylamine (0.67 mL, 6.8 mmol, 400 eq.). The reaction mixture was stirred for 10 min at room temprature, and then evaporated to dryness. The residue was recrystallizated from CH_2Cl_2-hexane to give faint yellow powder (10.7 mg). Yield 78 %.

^{1}H NMR (400 MHz, δ-value in $CDCl_3$) 11.03 (br, 4H, N*H*); 7.51 (t, 2H, J=8.1 Hz, pyridine-γ-*H*); 7.14 (d, 4H, J=8.1 Hz, pyridine-β-*H*); 5.19 (s, 2H, meso-*H*); 2.5~2.8 (m, 20H, CH_2Me and NHC$H_2$$CH_2CH_2CH_3$); 1.51 (qui, 4H, J=7.4 Hz, $NHCH_2CH_2CH_2CH_3$); 1.31 (se, 4H, J=7.4 Hz, $NHCH_2CH_2CH_2$Me); 1.19, 1.14 (t×2, 12H×2, J=7.4 Hz, CH_2*Me*); 0.87 (t, 6H, 7.4 Hz, $NHCH_2CH_2CH_2$*Me*). UV-vis (λ_{max} (log ε) in CH_2Cl_2) 309 (4.70), 356 (4.49) nm. Emission (λ_{max} in CH_2Cl_2 excited at 309 nm) 430 nm. ESI-MS 736.53/736.51 (found/calcd for $C_{52}H_{72}N_8+H^+–C_4H_9NH_2$). Analysis calcd. for $C_{52}H_{72}N_8•(H_2O)_4$: C, 70.87; H, 9.15; N, 12.72. Found: C, 70.90; H, 8.80; N, 12.86.

meso-Cyanodi-2,6-pyrihexaphyrin (**9a**)

A mixture of 11-cyano-11,33-dihydrodi-2,6-pyrihexaphyrin (**7a**) (15.4 mg, 23.2 μmol) and DDQ (6.4

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mg, 28.2 μmol, 1.3 eq.) in dry CH_2Cl_2 (5.9 ml) was stirred for 1 h at room temprature. The resulting solution was washed with aqueous K_2CO_3 solution, dried over Na_2SO_4, and then purified by column chromatography on alumina. The red fraction eluted with CH_2Cl_2 was evaporated to dryness and the residue was recrystallized from CH_2Cl_2-hexane to give red powder (7.5 mg). Yield 47 %.

^{1}H NMR (400 MHz, δ-value in $CDCl_3$) 12.24 (br, 1H, N*H*); 7.77 (t, 2H, J=7.9 Hz, pyridine-γ-*H)*; 7.36, 7.33 (d×2, 2H×2, J=7.9 Hz, pyridine-β-*H*); 6.92 (s, 1H, meso-*H*); 2.97, 2.66, 2.53, 2.47 (q×4, 4H×4, J=7.4 Hz, C*H*$_2$Me); 1.28, 1.21, 1.06, 1.01 (t×4, 6H×4, J=7.3~7.6Hz, CH_2*Me*). UV-vis (λ_{max} (log ε) in CH_2Cl_2) 290 (4.52), 330 (sh, 4.33), 469 (4.78), 585 (sh, 3.80). ESI-MS 688.92/688.41 (found/calcd for $C_{45}H_{49}N_7$+H^+). Analysis calcd. for $C_{45}H_{49}N_7$: C, 78.57; H, 7.18; N, 14.25. Found: C, 78.45; H, 7.32; N, 14.23.

meso-Dicyanodi-2,6-pyrihexaphyrin (**10a**)

A mixture of di-2,6-pyrihexaphyrin (**5a**) (20 mg, 30.2 μmol), trimethylsilyl cynaide (16.8 μL, 132.9 μmol, 4.4 eq.), and tetra-n-butylammonium fluoride (1.57 mg, 7.25 μmol, 0.24 eq.) in dry $CHCl_3$ (10 ml) was stirred for 18 h at room temperature under argon. DDQ (14.5 mg, 63.9 μmol, 2.2 eq.) was then added and the reaction mixture was stirred for 1 h at room temprature. The resulting solution was neutralized with aqueous K_2CO_3 solution. The separated organic layer was dried over Na_2SO_4 and then purified by column chromatography on alumina. The red fraction eluted with CH_2Cl_2 was evaporated to dryness and the residue was recrystallized from CH_2Cl_2-hexane to give red powder (15.5 mg). Yield 72 %.

^{1}H NMR (400 MHz, δ-value in $CDCl_3$) 12.40 (br, 2H, N*H*); 7.81 (t, 2H, J= 8.1 Hz, pyridine-γ-*H)*; 7.37 (d, 4H, J=8.1 Hz, pyridine-β-*H*); 2.97, 2.47 (q×2, 8H×2, J=8.3 Hz, C*H*$_2$Me); 1.28, 1.02 (t×2, 12H×2, J=7.6 Hz, CH_2*Me*). UV-vis (λ_{max} (log ε) in CH_2Cl_2) 291 (4.54), 319 (sh, 4.42), 497 (4.74), 568 (sh, 4.03) nm. ESI-MS 713.25/713.41 (found/calcd for $C_{46}H_{48}N_8$+H^+). Analysis calcd. for $C_{46}H_{48}N_8$•(H_2O): C, 75.59; H, 6.89; N, 15.33. Found: C, 75.91; H, 6.91 ; N, 15.15.

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2. ^{1}H NMR spectra.

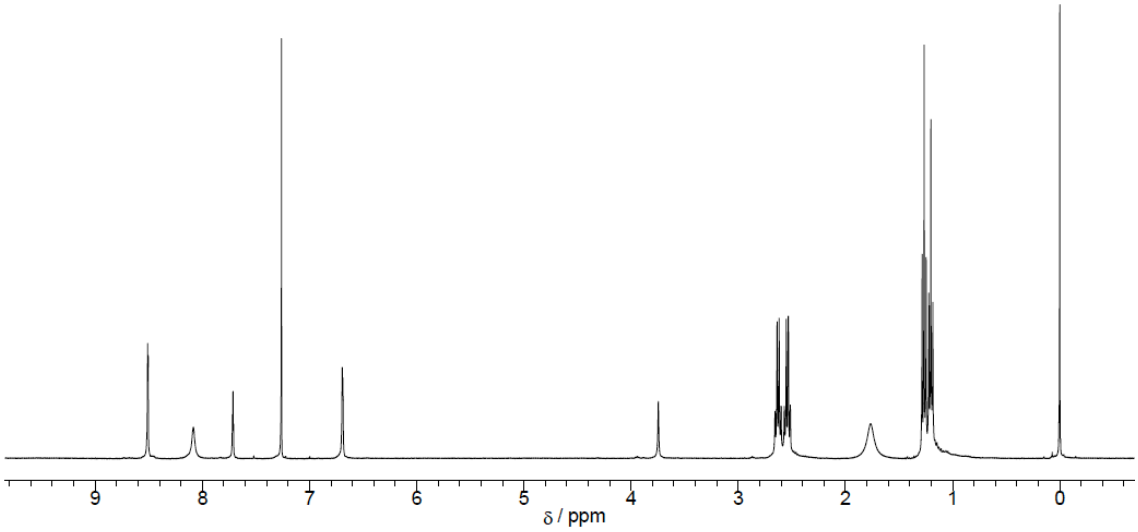

Figure S1. ^{1}H NMR spectrum of **4b** in $CDCl_3$.

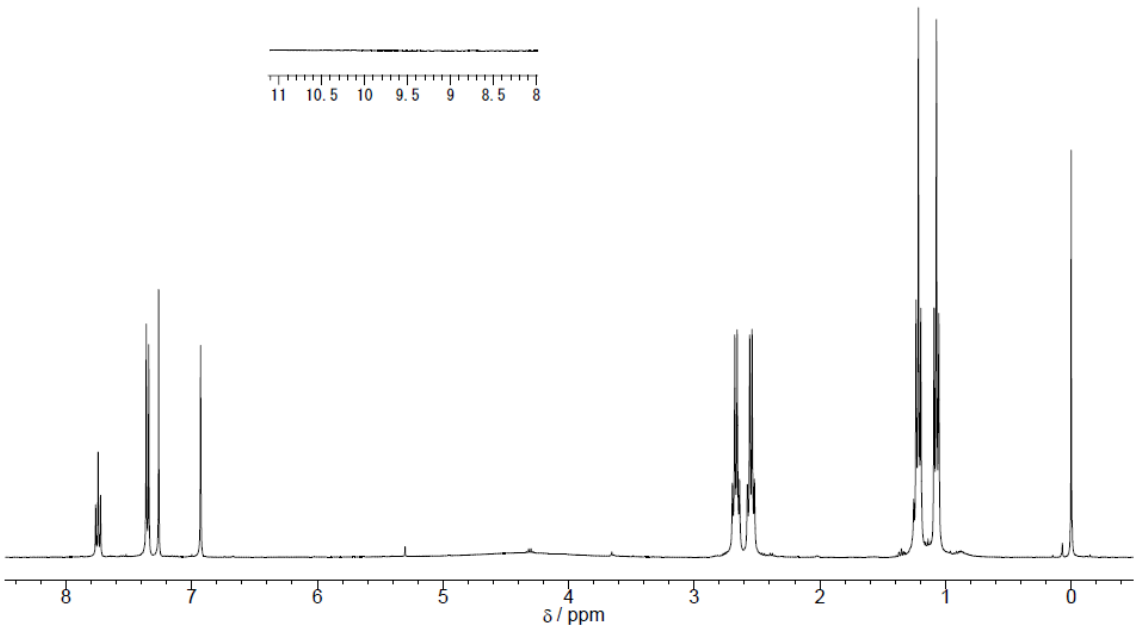

Figure S2. ^{1}H NMR spectrum of **5a** in $CDCl_3$.

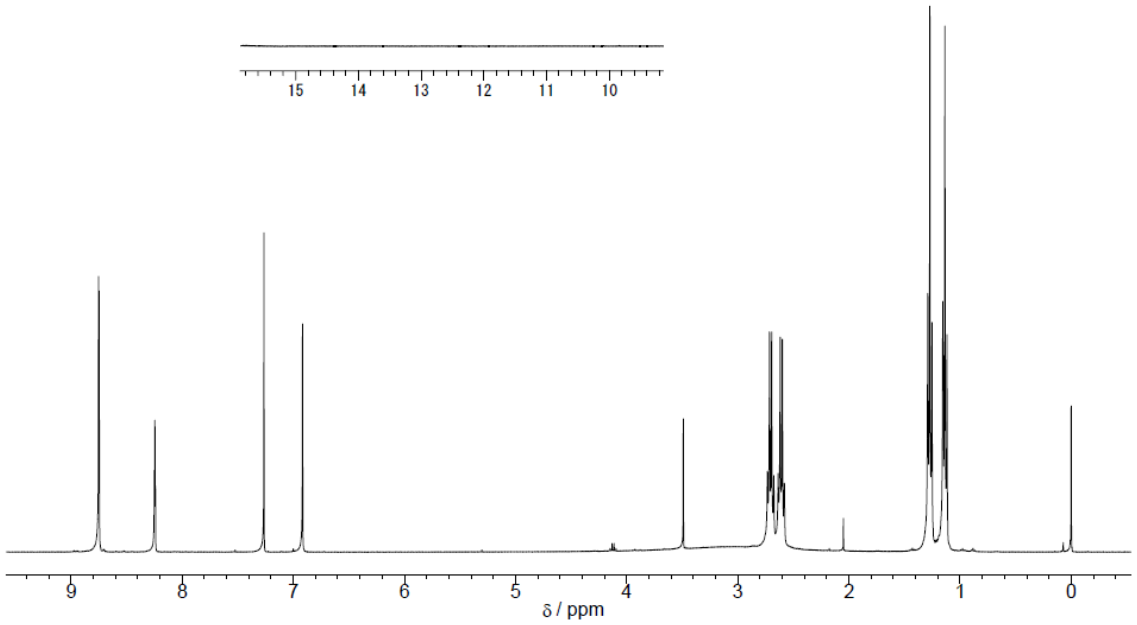

Figure S3. ^{1}H NMR spectrum of **5b** in $CDCl_3$

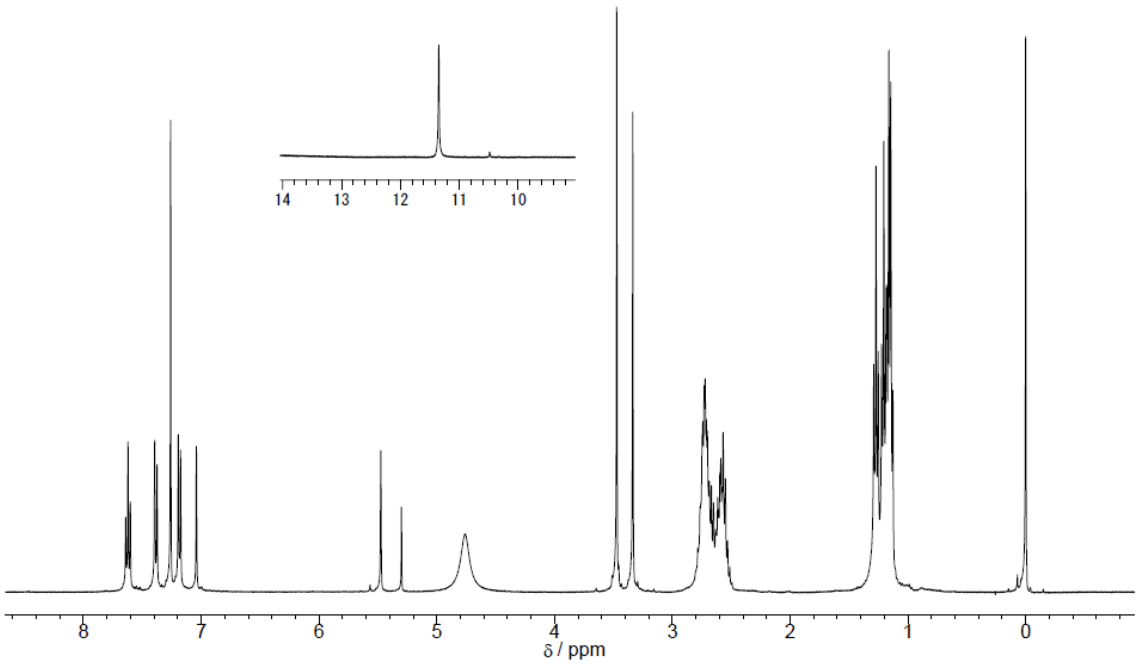

Figure S4. ^{1}H NMR spectrum of **6a** in $CDCl_3$.

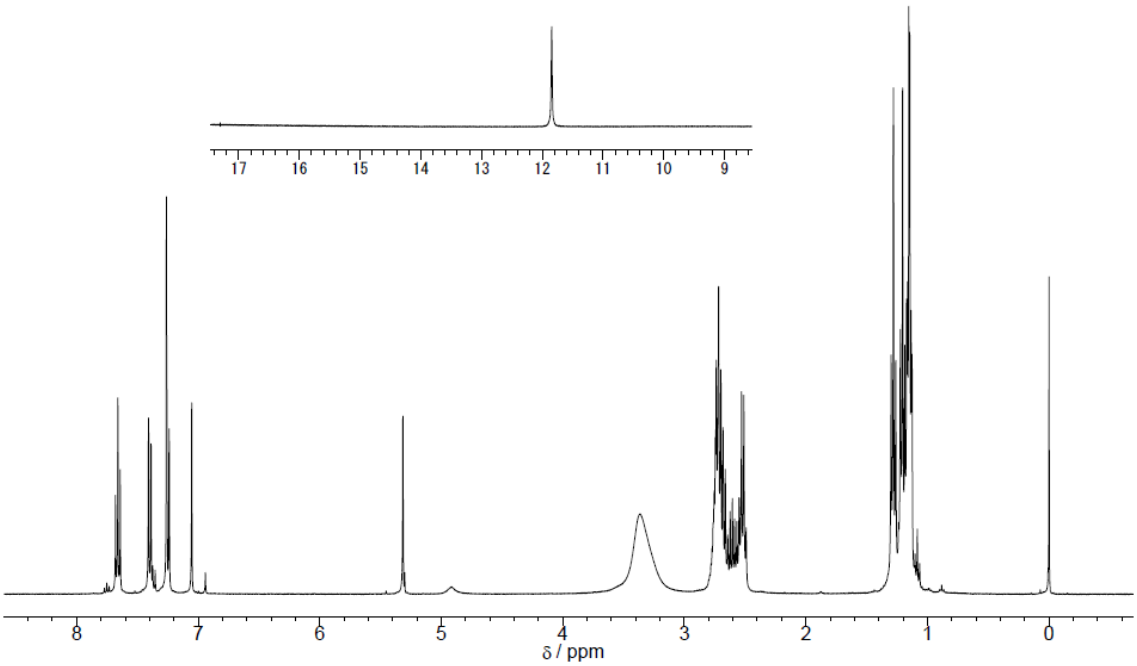

Figure S5. ^{1}H NMR spectrum of **7a** in $CDCl_3$.

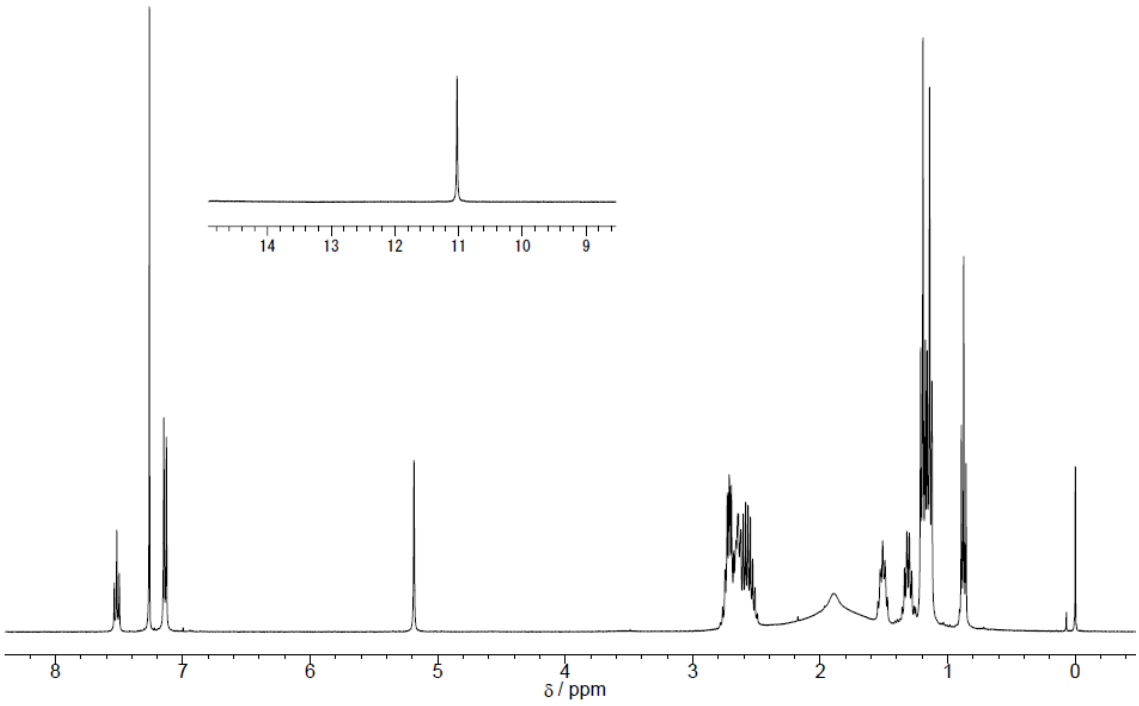

Figure S6. ^{1}H NMR spectrum of **8a** in $CDCl_3$.

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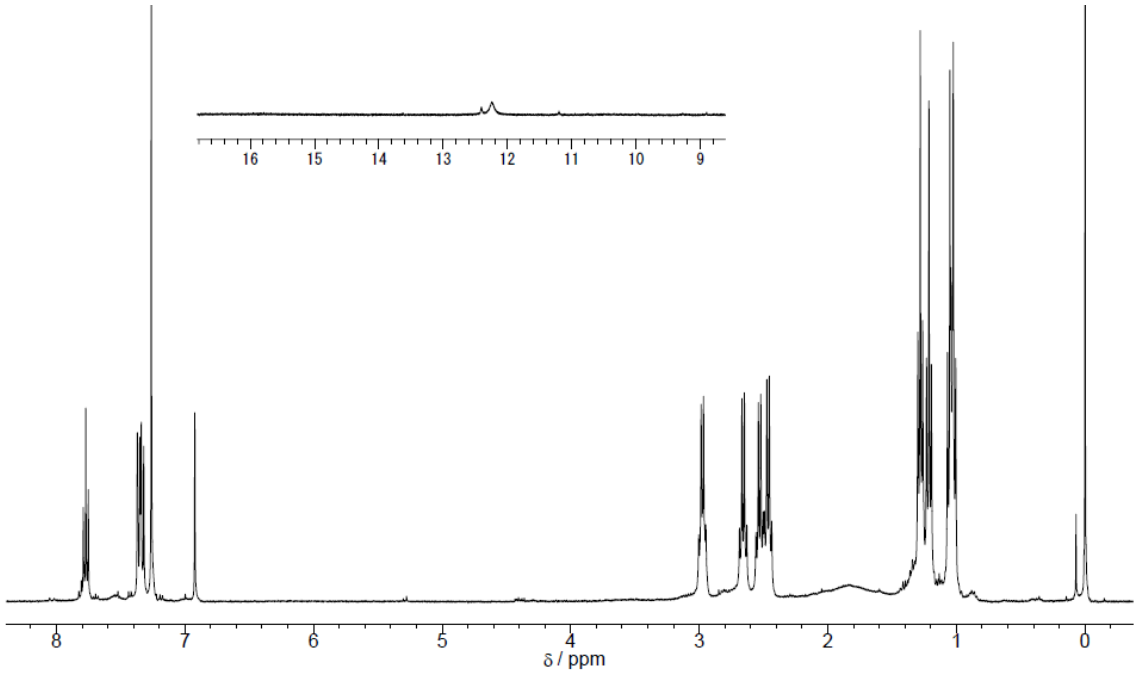

Figure S7. ^{1}H NMR spectrum of **9a** in $CDCl_3$.

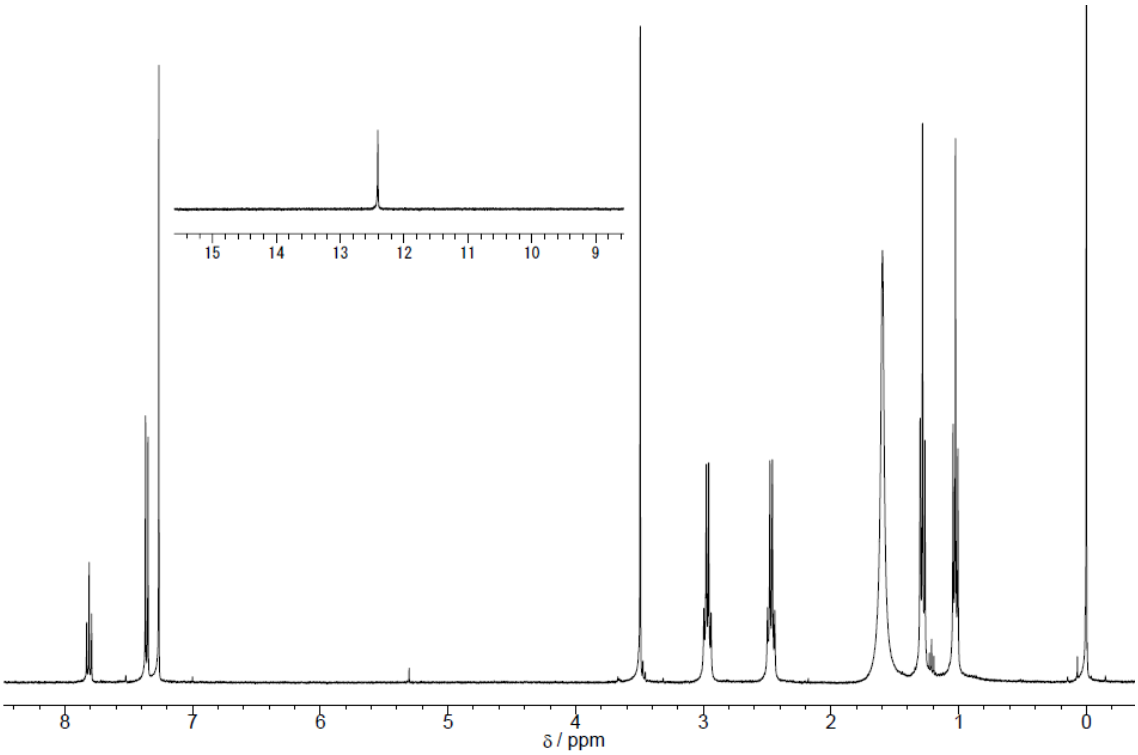

Figure S8. ^{1}H NMR spectrum of **10a** in $CDCl_3$.

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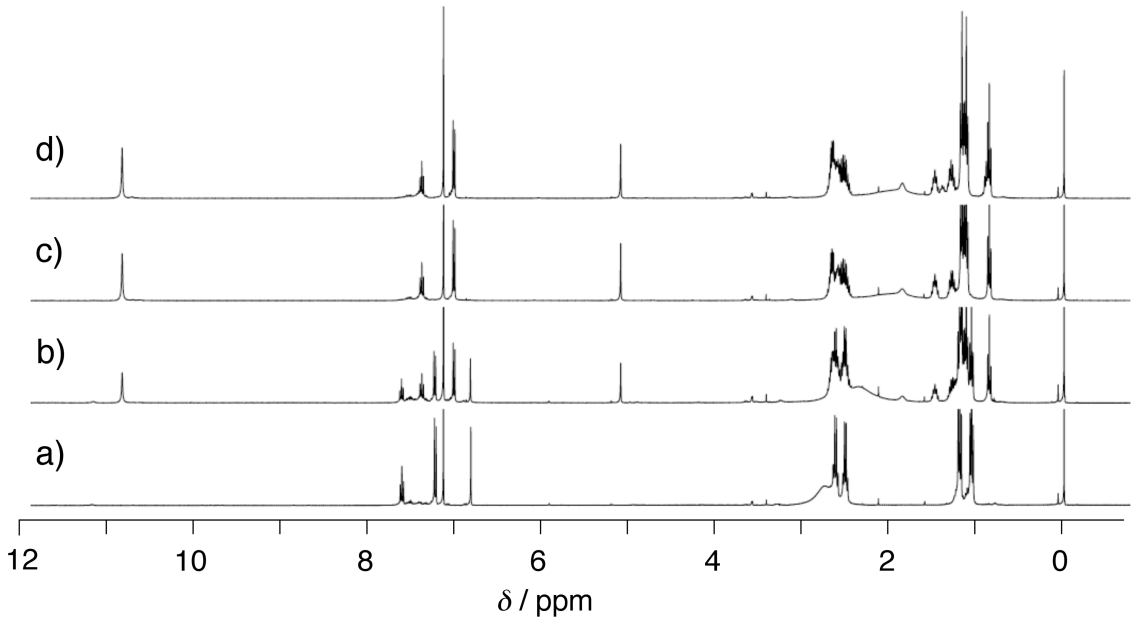

Figure S9. ^{1}H NMR titration of **5a** with butylamine in $CDCl_3$; a) 0 equiv, b) 1.0 equiv, c) 2.0 equiv, d) 3.0 equiv of butylamine

3. X-ray crystallography

Crystallographic data for **5a**, **5b**, **6a**, **7a** and **10a**

Crystals suitable for X-ray diffraction was obtained by recrystallization using a combination of hexane-CH_2Cl_2 or methanol-CH_2Cl_2. Bruker Apex-II diffractometer equipped with a CCD detector was used for data collection. An empirical absorption correction was applied using the SADABS program. The structure was solved by the direct method and refined by full-matrix least-squares calculations on F^2 using the Shelxtl 97 program package.[*)] All the hydrogens inside the cavity were taken from the difference Fourier map and fully refined.

[*)] Sheldrick, G. M. SHELXTL 5.10 for Windows NT: Structure Determination Software Programs; Bruker Analytical X-ray Systems, Inc., Madison, WI, 1997.

Crystallographic data of **5a** (from CH_2Cl_2/n-hexane): $C_{44}H_{50}N_6$•(H_2O), M = 680.92, triclinic, *P-1*, a = 10.242(3), b = 13.946(4), c = 15.390(5) Å, α = 114.240(4)°, β = 95.139(4)°, γ = 103.687(4)°, V = 1903.7(9) Å^3, Z = 2, D_{calc} = 1.188 Mg/m^3, μ(Mo-Kα) = 0.072 mm^{-1}, T = 296(2) K, R_1 = 0.0530, wR_2 = 0.1385, GOF (on F^2) = 0.865 ($I > 2.00\sigma(I)$), CCDC 865639. **5b** (from CH_2Cl_2//n-hexane): $C_{44}H_{50}N_6$, M = 662.90, monoclinic, *P2(1)/n*, a = 13.435(3), b = 18.486(3), c = 16.141(3) Å, α = 90.00°, β = 108.757(2)°, γ = 90.00°, V = 3795.9(12) Å^3, Z = 4, D_{calc} = 1.160 Mg/m^3, μ(Mo-Kα) = 0.069 mm^{-1}, T = 296(2) K, R_1 = 0.0442, wR_2 = 0.1253, GOF (on F^2) = 0.869 ($I > 2.00\sigma(I)$), CCDC 865640. **6a** (from CH_2Cl_2//MeOH): $C_{45}H_{54}N_6O$•(MeOH), M = 694.94, monoclinic, *P2(1)/c*, a = 10.338(2), b = 16.272(3), c = 24.880(5) Å, α = 90.00°, β = 97.340(3)°, γ = 90.00°, V = 4151.1(14) Å^3, Z = 4, D_{calc} = 1.112 Mg/m^3, μ(Mo-Kα) = 0.068 mm^{-1}, T = 296(2) K, R_1 = 0.0528, wR_2 = 0.1532, GOF (on F^2) = 1.064 ($I > 2.00\sigma(I)$), CCDC 865641. **7a** from (CH_2Cl_2//MeOH): $C_{45}H_{51}N_7$•(MeOH)(CH_2Cl_2), M = 806.90, triclinic, *P-1*, a = 11.025(3), b = 14.059(4), c = 15.430(4) Å, α = 71.589(4)°, β = 86.948(4)°, γ = 77.737(4)°, V = 2217.3(11) Å^3, Z = 2, D_{calc} = 1.209 Mg/m^3, μ(Mo-Kα) = 0.190 mm^{-1}, T = 296(2) K, R_1 = 0.0604, wR_2 = 0.1524, GOF (on F^2) = 0.856 ($I > 2.00\sigma(I)$), CCDC 865642. **10a** (from CH_2Cl_2//n-hexane): $C_{46}H_{48}N_8$, M = 712.92, monoclinic, *P2(1)/n*, a = 17.110(4), b = 10.863(2), c = 22.611(5) Å, α = 90.00°, β = 105.459(3)°, γ = 90.00°, V = 4050.7(15) Å^3, Z = 4, D_{calc} = 1.169 Mg/m^3, μ(Mo-Kα) = 0.071 mm^{-1}, T = 296(2) K, R_1 = 0.0457, wR_2 = 0.1308, GOF (on F^2) = 0.930 ($I > 2.00\sigma(I)$), CCDC 865643.

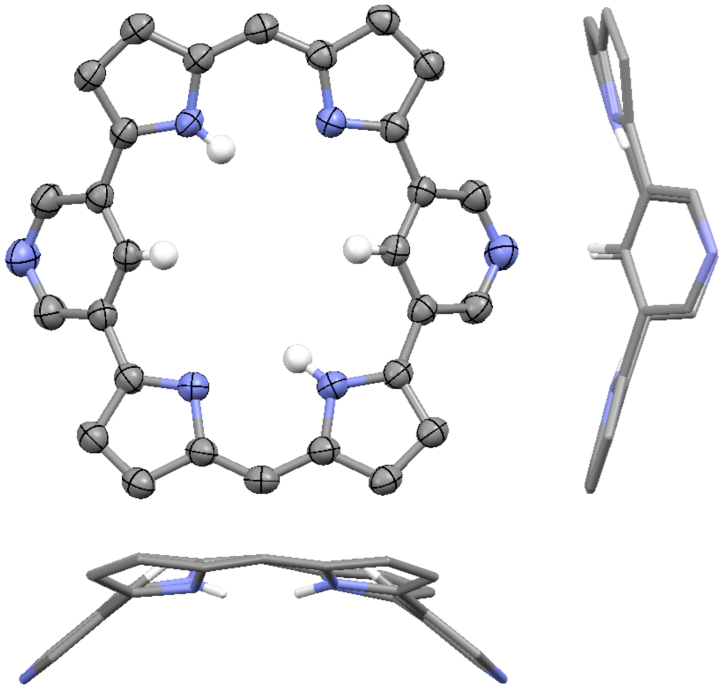

Figure S10. X-ray structures viewed along right axes of **5b** with the 50% probability level thermal ellipsoids. Peripheral ethyl groups are omitted for clarity and the NH and OH hydrogens appeared in the Fourier map and fully refined.

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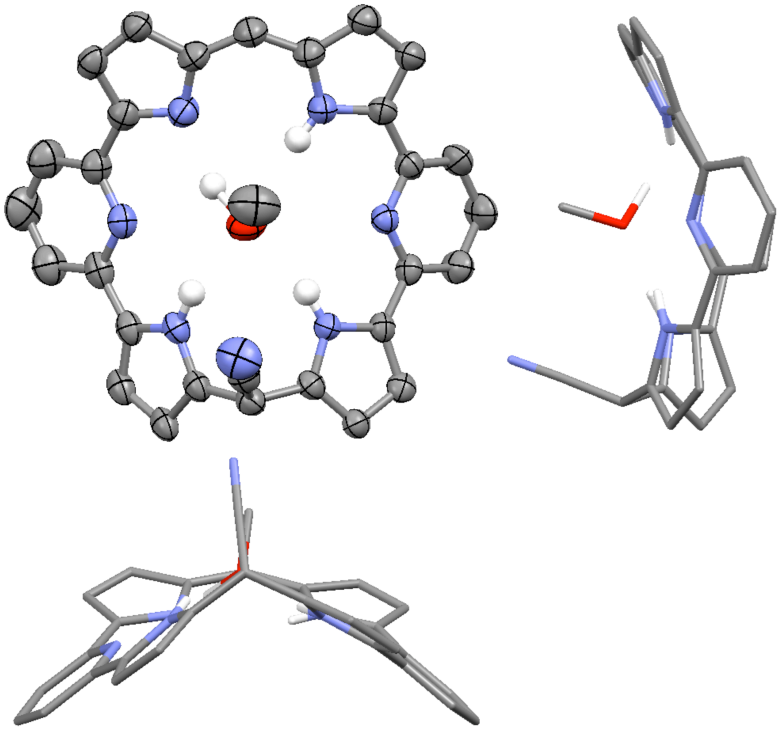

Figure S11. X-ray structures viewed along right axes of **7a** with the 50% probability level thermal ellipsoids. Peripheral ethyl groups are omitted for clarity and the NH and OH hydrogens appeared in the Fourier map and fully refined.

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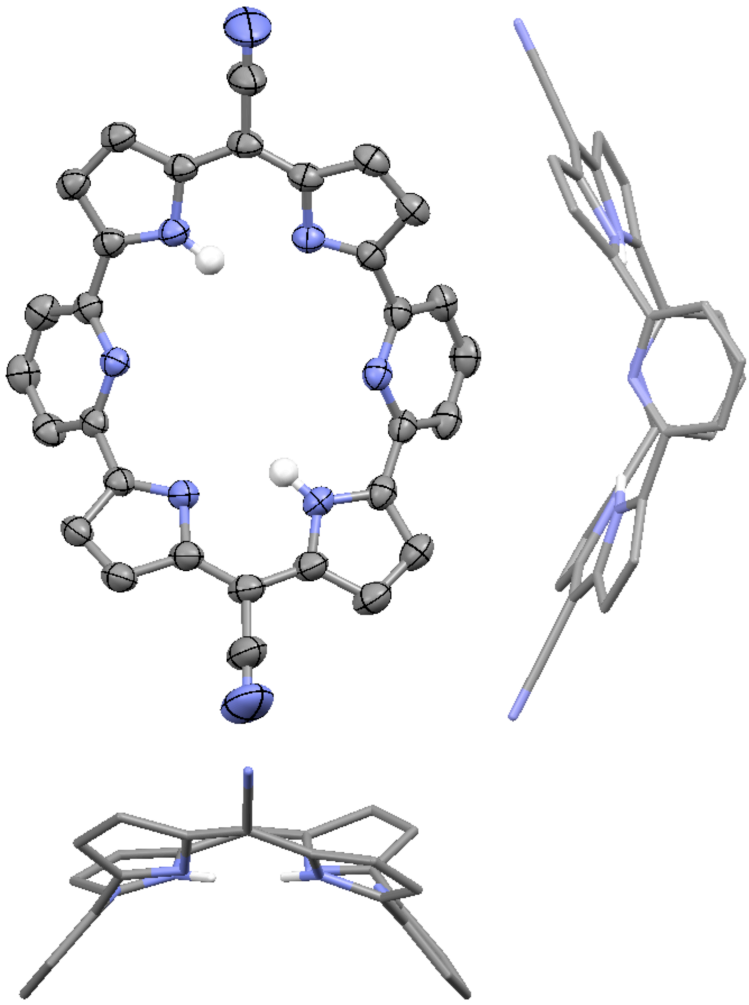

Figure S12. X-ray structures viewed along right axes of **10a** with the 50% probability level thermal ellipsoids. Peripheral ethyl groups are omitted for clarity and the NH and OH hydrogens appeared in the Fourier map and fully refined.